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"Adhesion Measurements of Thin Films in Corrosive Environments"

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Introduction

With the use of thin films reaching a wide variety of applications, it is important to know how thin films will hold up in a variety of environmental conditions. Here, water effects on thin film adhesion were studied on copper and diamond like carbon (DLC) films. Regardless of the film being considered, it is usually advantageous to have a thin film that adheres well to its substrate. Because of the potentially harsh environments that thin films may be introduced to during their processing or use, the effect of water on thin film adhesion need to be further studied.

There are several methods currently being used to measure the adhesion of thin films which include the following: four-point bend test, indentation, scratch and pull off tests. Here we used the superlayer indentation test for adhesion measurements in the dry environment. For measuring film adhesion in a wet environment a modified version of the superlayer nanoindentation test was developed. Quantitative adhesion tests yield the practical work of adhesion, or strain energy release rate, G, value. When G is greater than the material's resistance to crack growth, Γ, crack propagation will occur. The formerly stated is the Griffith criterion and can also be applied to interfacial fracture.

$$G > \Gamma_i$$

For the indentation tests, as the depth and load of the indents increase a steady state strain energy release rate value will be observed. The steady state strain energy release rate can be equated to the film's adhesion to the substrate. This steady state strain energy release rate will appear as a leveling or plateau in the indentation data when G is plotted versus the delamination blister radius to indenter tip contact radius ratio, x/a (Figure 1).

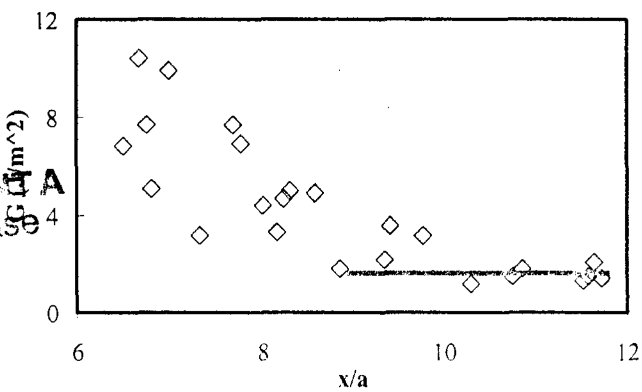


Figure 1. Steady state strain energy release rate.

After indentation test were performed in both dry and wet environments, the results were compared and larger delamination blister radius was immediately noticed and a significant lowering in film adhesion was measured when water was able to reach the film/substrate interface. But when water was not allowed to reach the interface, no changes in film adhesion were observed.

Samples Description

There were a total of five samples tested, two copper films of different thickness and three samples supplied by Seagate which had different thicknesses of DLC and magnetic layers. Copper adhesion has been of interest since the late 1990's when research was conducted regarding the possibilities of replacing aluminum with copper as the choice for interconnect material in microelectronics. Not only does copper have better electrical and thermal conductivity than aluminum, copper interconnects also provide higher current densities and better electromigration performance. One major disadvantage, however, of using copper films is that they have lower adhesion to Si substrates than aluminum. For this reason, it was necessary to quantify the adhesion of copper to silicon and to experiment with moisture effects that may be seen in a production environment.

Seagate has been testing the amorphous carbon films as a potential protective layer on the surface of magnetic media such as read and write heads in hard drives. DLC films are being used due to their chemical inertness, high hardness and wear resistance with low friction coefficients [1, 2]. Since high adhesion of DCL films is required, any delamination of the coating from the surface can damage the head and make media unreadable.

The two copper films were sputter deposited on a silicon substrate with a 1 μm layer of SiO_2. The copper layer was covered by a 1.1 μm thick tungsten superlayer which was sputter deposited, having a compressive residual stress of 320 MPa. The first copper film tested was 67 nm thick, and the second was 97 nm thick.

The DLC films were deposited by means of PECVD on 3" silicon wafers with a 300 nm thick layer of SiO_2. An 800 nm thick tungsten superlayer was sputter deposited on top of the DLC films with 1.9 GPa compressive residual stress.

Experimental Procedure

Nanoindentation is a very successful way for measuring the elastic modulus and hardness of thin films, but it can also be used to measure thin film adhesion [3]. Delamination and crack growth are induced with the combination of indentation stresses and residual stresses present in the thin film. In Marshall and Evans' analysis of delamination blisters created by indentation, they treated the section of film above the delaminating crack as a rigidly clamped disc [4]. There are two problems likely to be encountered with the single layer indentation test, which include pile-up of the thin film around the indenter tip and penetration to depths greater than the film thickness. If the indent is made too deeply, deformation and cracking of the substrate may occur and will reduce the validity of the test. Both problems can be controlled using the superlayer indentation technique, which consists of depositing a hard superlayer on top of the film of interest. The superlayer can be deposited by means of a relatively low temperature physical vapor technique such as sputtering. In sputter deposition the temperature is not

high enough to alter the microstructure or the interface of the original film. The superlayer can be tailored to optimize conditions for film thickness and residual stress, which allows for greater driving force for the same penetration depth to film thickness ratio. One condition that must be met for the superlayer indentation method to work is that the superlayer must adhere to the film more strongly than the film adheres to the substrate. If this condition is not met, the measurement obtained for adhesion will be for the superlayer to the film and not the adhesion of the film of interest to the substrate.

Kriese and Gerberich have combined the idea of a superlayer test with the Marshall and Evans findings by applying the laminate theory in order to calculate the strain energy release rate for a multilayer sample [5]. In most cases the superlayer is much thicker than the underlayer, so the laminate theory does not need to be applied and the test can be treated as the single layer test defined by Marshall and Evans. The following equation takes into account the indentation stress σ_I, residual stress σ_R, and critical buckling stress σ_B of the film to calculate the strain energy release rate G [4]:

$$G = \frac{h\sigma_I^2\left(1-\nu_f^{\,2}\right)}{2E_f} + (1-\alpha)\frac{h\sigma_R^2\left(1-\nu_f\right)}{E_f} - (1-\alpha)\frac{h(\sigma_I-\sigma_B)^2\left(1-\nu_f\right)}{E_f}$$

where h, υ, and E are the film thickness, Poisson ratio and elastic modulus respectively. In order to calculate the indentation and buckling stresses, needed for obtaining the film adhesion, the delamination blister radius and plastic indentation depth must be known. Delamination blister radius can be measured with an optical microscope and the plastic indentation depth can be extracted from the load-displacement curve recorded during indentation. Figure 2 has been captured with an optical microscope and shows the delamination blister radius and indenter tip contact radius labeled x and a respectively. In the load-displacement curve the plastic indentation depth is extrapolated labeled with the symbol δ.

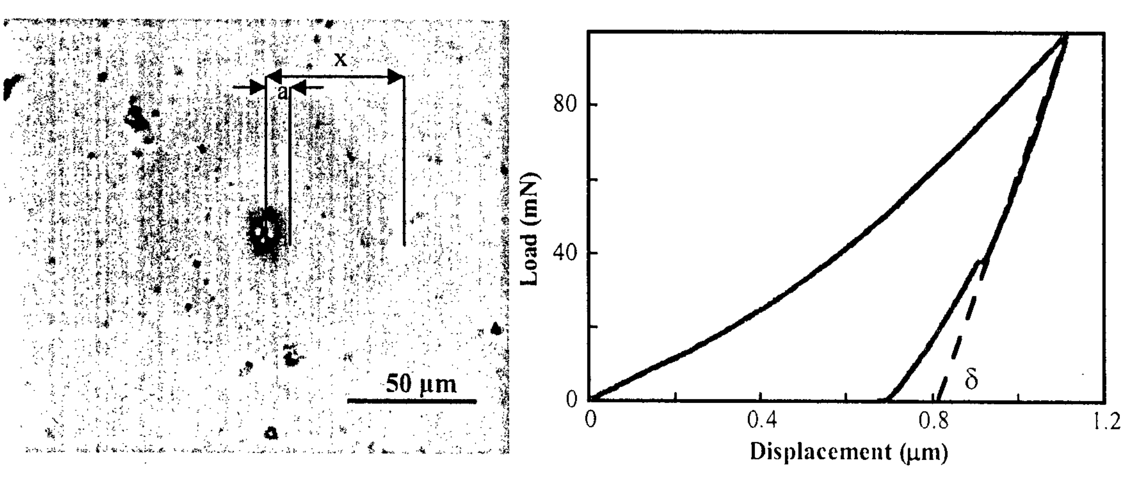
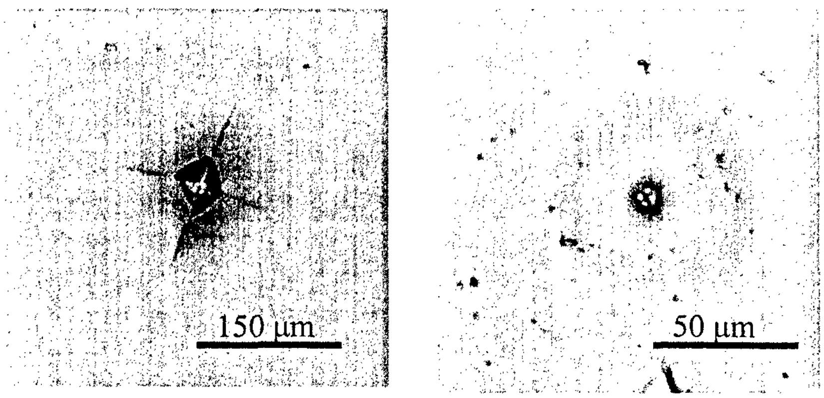


Figure 2. Data retrieved from indentation test.

In order to calculate film adhesion in a wet environment, modifications were made to the superlayer indentation test by adding an introductory indent so that water could be introduced to the film/substrate interface. The introductory indent was made to a depth equal to that of the film thickness, then the tip was removed from the film and water was placed with a pipette in the location of the introductory indent. After water was introduced a second indent was performed in the same spot as the introductory one to further delaminate the film and calculate film adhesion in the wet environment. As with the superlayer indentation test, plastic indentation depth and delamination blister radius were needed to calculate adhesion. Delamination blister resulting from both wet and dry indents can be compared in Figure 3. Delamination blister radius in a wet environment was significantly larger than delamination blister radius in a dry environment for indents made to the same maximum load. Radial film cracks and asymmetrical blister shape were also observed more frequently in a wet environment than in a dry environment.



a) Wet indent b) Dry indent

Figure 3. Wet indent compared to dry indent.

Before the modified superlayer indentation test results in a wet environment were compared to the normal superlayer indentation test results, the modified superlayer indentation test was performed in a dry environment and compared to results from the normal test in a dry environment. Modified test in a dry environment exhibited comparable results, proving that the modified test did not affect the adhesion measurements [6]. In Table 1 the results of the two indentation tests in a dry environment are compared. The same *x/a* ratios were used to compare the two tests.

Table 1. Modified indentation test compared to the normal test in a dry environment.

Indent Procedure	x/a	G (J/m^2)
Double Indent (Dry)	7 - 8	7.8 ± 0.3
Single Indent (Dry)	7 - 8	8.1 ± 0.5

When analyzing the data from the modified indentation test, the load-displacement curve from the second indent had to be shifted to account for the depth of

the introductory indent. The reason for this is that the Hysitron Triboindenter that was used for the testing recorded the first point of contact as zero displacement. In Figure 4 load-displacement curves from the single indent test and modified test were compared. The open circle and triangle traces are from the modified test (introductory and the second indents respectively). The open square trace is an indent from the normal superlayer indentation test.

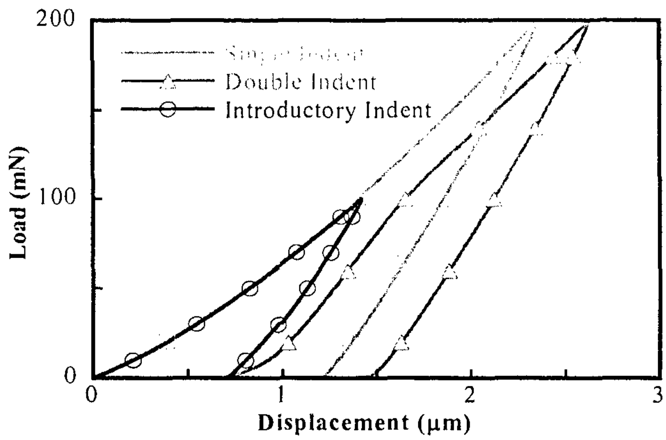


Figure 4. Load-displacement results in a dry environment comparing both tests.

Adhesion Results

The most important factor in producing larger delamination blisters and significantly lowering film adhesion was that water had to reach the film/substrate interface. When that happened, Cu adhesion was reduced by a factor of about 20 and for the different Seagate samples film adhesion reduced by a factor between 3 and 50. However, when water did not reach the interface there were no changes observed in film adhesion. Since the indentation tests are localized tests, repeated indents to a range of loads and locations on the wafers were performed to obtain average adhesion values for the different samples. The adhesion measurements for both wet and dry indents are presented in Table 2.

Table 2. Adhesion results comparing wet and dry environments.

Sample	Adhesion Dry (J/m^2)	Adhesion Wet (J/m^2)
Cu 67 nm	2.74	0.15
Cu 97 nm	1.98	0.10
CHx 5 nm	2.23	0.04
CHx 20 nm & CoCrPtTa 20 nm underlayer	2.02	0.66
CoCrPtTa 20 nm	2.03	0.16

Figure 5 compares the load-displacement curve of a single dry indent with the two indents conducted in a wet environment. As discussed earlier the second indent in the modified indentation test had to be shifted to account for the final indentation depth of the introductory indent.

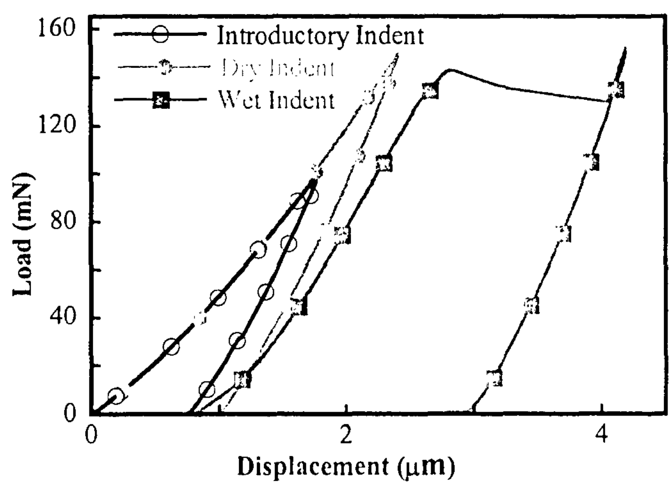


Figure 5. Load-displacement curves of wet and dry indents.

Delamination Channels

Since the Seagate samples had a much larger amount of residual stress present in the tungsten superlayer than the copper samples, straight sided and telephone cord delaminations were observed when a mechanical force was applied or when water was introduced to the interface. Delamination channels were formed by applying a mechanical force through scribing the wafers, piercing them with a microprobe tip or performing indents to large loads. If small delaminations were present on the edge of the wafer pieces, the addition of water at the edge would sometimes propagate delaminations. Depending on the amount of residual and buckling stress present in thin films, different delamination morphology was seen. Examples of both straight sided and telephone cord delaminations can be seen in Figure 6.

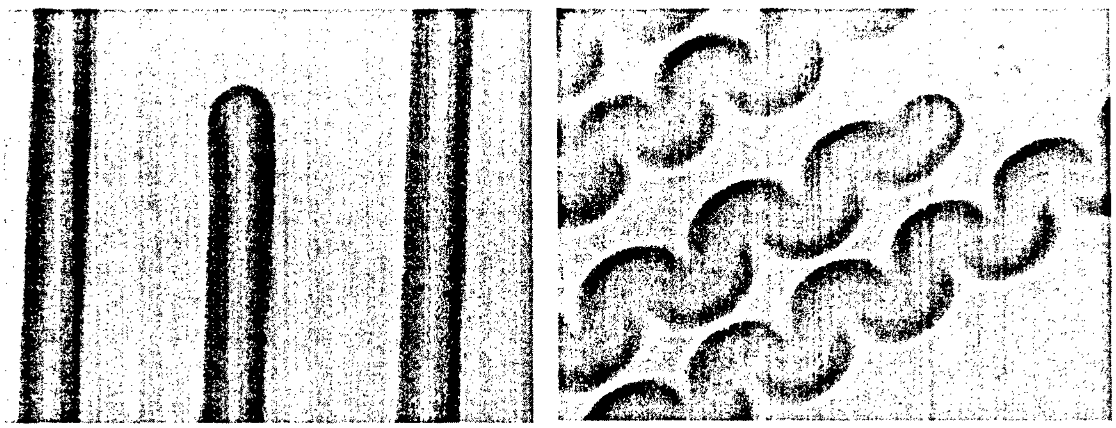


Figure 6. Straight sided and telephone cord delaminations.

It has been demonstrated that for residual to buckling stress ratio of $\sigma_r/\sigma_B = 5$, straight-sided or Euler blisters will be produced, whereas $\sigma_r/\sigma_B = 6.5$ predicts bumps or varicose blisters, and for $\sigma_r/\sigma_B = 7.5$ telephone cord blisters would appear [7, 8]. These ratios were found on a microscopic level by depositing a highly stressed superlayer on a substrate that had patterning of low interface adhesion areas surrounded by regions of high adhesion. The low adhesion areas were patterned in a tapered fashion so that the critical buckling stress could be varied while having a constant residual stress. On a macroscopic level the stresses were controlled by gluing a polycarbonate strip to a PVC block and applying forces in two directions using screws (perpendicular and parallel to the strip). In the latter experiment where the biaxial stress could be controlled, various buckling patterns appear when the stresses perpendicular and parallel to the film strip were not equal [8].

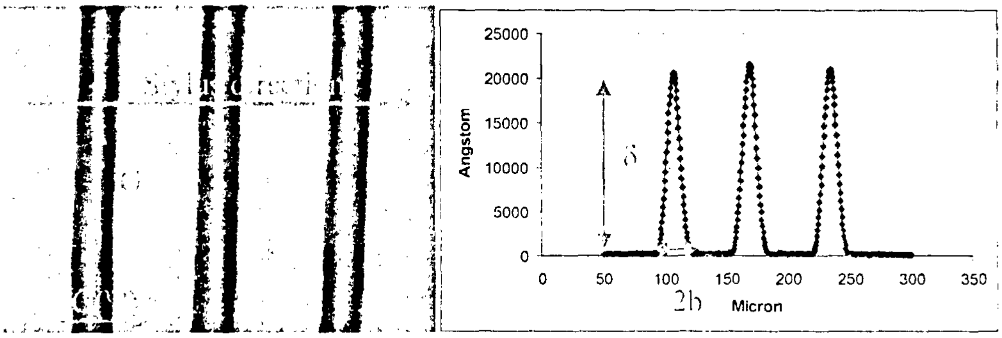


Figure 7. Straight sided delamination profiles.

By taking a profile of the straight sided delamination channels, film residual stress and adhesion could be calculated [9]. Analysis of the delaminations channels was based on findings by Hutchinson and Suo and then compared to the calculations for film adhesion by the Marshall and Evans method using nanoindentation. Using the Hutchinson and Suo developments the critical buckling stress, σ_B, can be calculated knowing the delamination blister half width b, film Modulus E, Poisson ratio v and film thickness h. After the critical buckling stress has been solved for the residual stress, σ_r, in the film can be calculated by measuring the delamination blister height δ. Both the delamination blister height and width were measured using a profilometer. With both the critical buckling and residual stress known the strain energy release rate in the buckling and steady state direction can be solved for. Equations used for the previously mentioned are listed below [9].

$$\sigma_B = \frac{\pi^2}{12}\frac{E}{(1-\nu^2)}\left(\frac{h}{b}\right)^2$$

$$\sigma_r = \frac{3}{4}\sigma_B\left(\frac{\delta^2}{h^2}+1\right)$$

$$G(\Psi) = \frac{(1-\nu^2)h}{2E}(\sigma_r - \sigma_B)(\sigma_r + 3\sigma_B)$$

$$G_{SS} = \frac{(1-\nu^2)h\sigma_r^2}{2E}\left(1-\frac{\sigma_B}{\sigma_r}\right)^2$$

After the straight-sided delamination profiles were obtained, the strain energy release rate in the buckling direction was solved to be approximately the same as the strain energy release rate obtained from the nanoindentation test for film adhesion.

Discussion

The lowering of film adhesion with the presence of water can be attributed to a chemical reaction at the crack tip which is assisting in bond rupture. Literature shows strong crack velocity dependence on relative humidity for ceramics, bulk glasses and metal/SiO_2 interfaces [10-13]. In those studies crack velocity was reported as a function

of applied load in environments with varying relative humidity. Three distinct regions could be seen, where the first two depend on the reaction rate of water at the crack tip and the rate of water transport to the crack tip. The system used in this study is similar in terms of the increasingly applied load driving crack propagation, but the crack velocity was not measured during indentation. For the indentation tests, instead of possible increased crack velocity, larger delamination blisters were observed. As the indenter tip is driven into the sample forcing film buckling and delamination from the substrate, water is provided a large enough channel to reach the crack tip. Along with indentation, capillary forces could be assisting in the transport of water to the crack tip.

Another significant effect of water reaching the interface could be due to lowering surface energies of the newly-formed surfaces at the crack tip. Lower surface energies would result in a lowering of the true work of adhesion:

$$W_A = \gamma_f + \gamma_s - \gamma_{fs}$$

where γ_f is the surface energy of film, γ_s is the surface energy of the substrate and γ_{fs} is the interfacial energy. This could be a reason for larger indentation blisters in the wet environment, as well as immediate delamination propagation for the W/DLC films induced by water.

After the telephone cord delaminations were observed and the water was removed from the samples, tape was applied to the top of the sample and then pulled to remove the film from the substrate. Outlines of the telephone cord delaminations were observed on the substrate and the removed film. A profilometer was then used to trace a surface profile of the outlines on the substrate film. The outlines observed were between 60 and 100 nm in height compared to the rest of the substrate. Previous studies found similar outlines from circular delamination blisters created by indentation tests. The studies referred to them as crack arrest fiducial marks and that they were formed when hydrocarbons are sucked into the crack tip during delamination [14, 15]. The fiducial marks in the previous study were measured to be from 5 to 15 nm in height. The authors were then able to use the substrate fiducial marks to back calculate film adhesion with either an elastic of plastic analysis by knowing the fiducial mark height and half width. Here it is speculated that the fiducial marks are much larger due to the larger amounts of debris being available to be sucked into the crack tip with the presence of water. A picture and profile of the fiducial marks observed on the substrate are shown in Figure 8 and the fiducial marks on the underside of the removed film are shown in Figure 9.

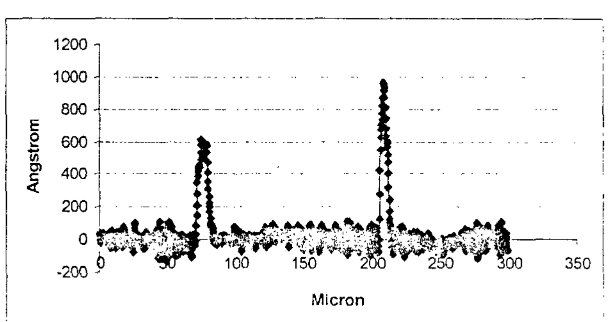


Figure 8. Fiducial mark profiles on a substrate.

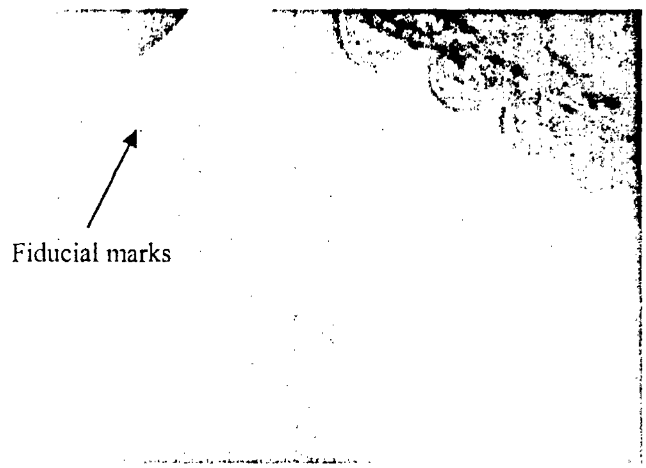


Figure 9. Fiducial marks on underside of the removed film.

When performing the modified indentation test in a wet environment it was noticed that load excursions would appear on the second indent. In the dry environment load excursions for the copper samples were observed at approximately 275 mN, where in the wet environment load excursions appeared at a lower load of approximately 125 mN (Figure 10). The excursions consistently appeared at the same loads for both the dry or wet environment indents.

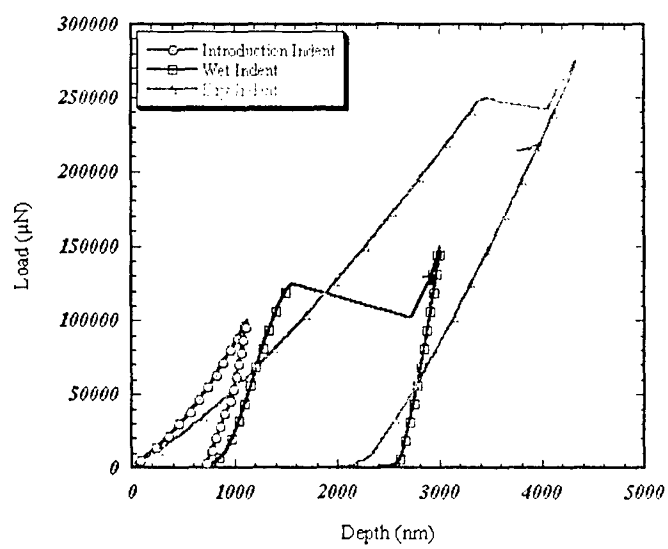


Figure 10. Load excursions in wet and dry indents.

Every time the load excursions have been observed, cracking of the buckled film and the substrate was observed. The radial cracks in the buckled thin film start at the indent in the center of the delamination blister and move outward. Fractures in the substrate were always observed at 90° angle from each other, corresponding top to the Si substrate crystal orientation. The blister profile is difficult to see Figure 11 due to the low focal depth necessary for observing substrate cracks. A conical tip with a one micron tip radius was used in all indentation tests.

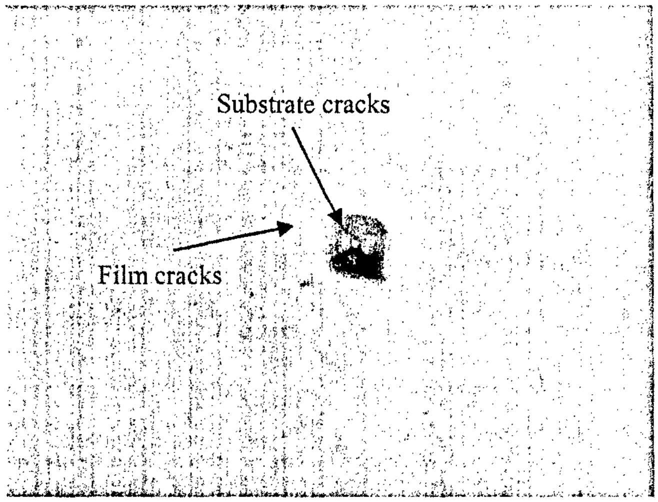


Figure 11. Radial cracks in the thin film and substrate.

Future Work

Up to this point it would be easy to think of a lowering in film adhesion as a negative outcome. But what if this phenomenon could be taken advantage of and turned into a positive outcome? It has been shown that water can propagate straight-sided and telephone cord delaminations at a rate up to ten microns per second (Figure 12).

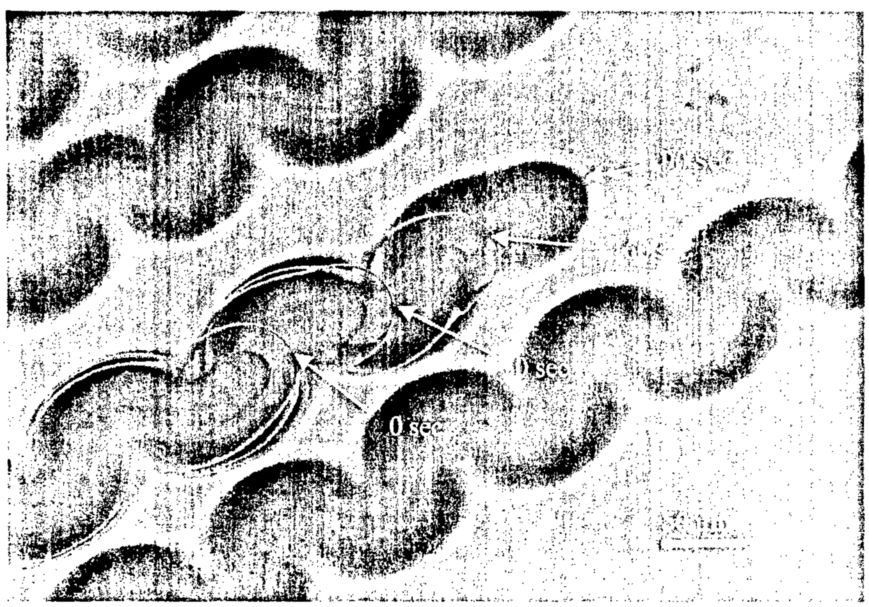


Figure 12. Telephone cord delamination propagation.

If the direction and size of the delaminations could be controlled, the delamination channels could possibly be used for microfluidic transport and mixing. With the use of manufacturing techniques common in the microelectronics industry this could be achieved. Current research is creating adhesion release layers so that

delamination location and size can be controlled. The release layers are strips of photoresist that act as a weak point for thin film stress relief. First a positive photoresist is spin-on-deposited, exposed and developed. Next a compressively stressed film of tungsten is sputter deposited (Figure 13). Finally, delaminations will be created along the resist layers if the tungsten is deposited with enough compressive stress. If the tungsten is deposited with small amounts of compressive stress, delamination will not occur until water or a mechanical force is applied.

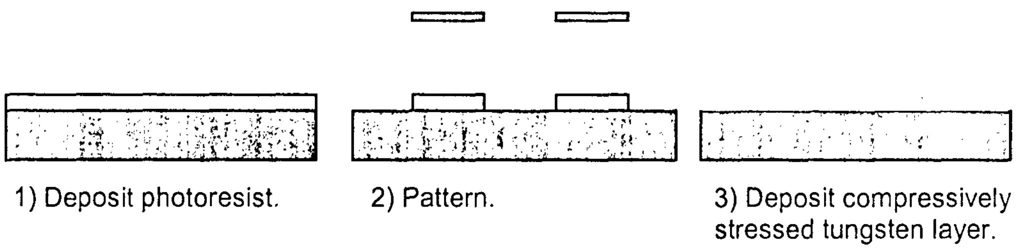


Figure 13. Patterning of adhesion release layers.

These delamination channels have the potential to be an inexpensive and non labor intensive way of creating microfluidic channels. Some current methods for creating microchannels are by dry etching into silicon or by creating molds and using PDMS with glass slides as the cover to the PDMS channels. Neither is necessarily hard, but both tend to be either costly or time consuming. With the use of a microprobe it has been observed that delamination channels can withstand a large amount of mechanical force before there's further delamination or fracture. A microprobe was used to push water back and forth through the delamination channels and can be observed in Figure 14. Even though this was a fairly crude experiment, it showed one possibility for forced fluid transport.

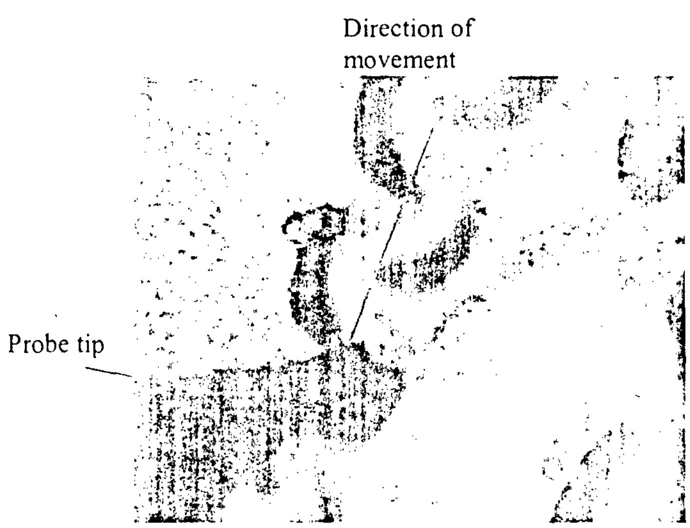


Figure 14. Manipulation of telephone cord delaminations with a microprobe tip.

By patterning larger elliptically-shaped areas of the adhesion release layer, delaminations can be created for fluid storage or fluid pumping if actuators were used. Figure 15 shows how larger patterned areas can be created for fluid storage or pumping, that can be controlled with a microprobe tip to force fluid flow.

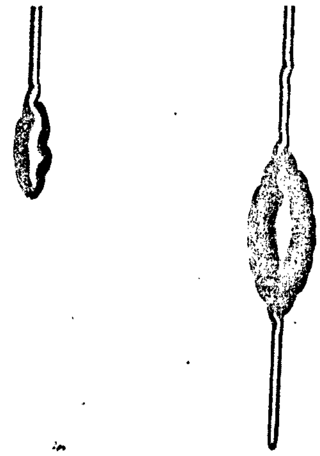


Figure 15. Delaminations for fluid storage and pump actuation.

Conclusions

A new test method was developed for measuring film adhesion in a wet environment by making modifications to the superlayer indentation test. When water was able to reach the film/substrate interface significant drops in film adhesion were observed. For the copper film samples tested adhesion dropped by a factor up to 20 and for the Seagate DLC samples a drop in adhesion was measured up to a factor of 50. When compressive residual stress was large enough in the films, the introduction of water to the interface resulted in straight-sided and telephone cord delamination propagation. The delamination propagation was measured up to tens of micron per second and proved to be a viable means for microfluidic transport.

Current and future work is focusing on utilizing water's effect on film adhesion in the area of microfluidics which could possible see use in the biomedical industry. Microelectronic manufacturing techniques are being used to create areas of lower film adhesion which will control delamination size and location.

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Appendix for the
Final Report for NACE Contract N000140210024

"Adhesion Measurements of Thin Films in Corrosive Environments"

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Ph.D. Graduate Student: Patrick Waters

The following presentations were made during the award period acknowledging NACE support:

1. *Thin Film Moisture Induced Delamination for Microchannel Fabrication*, P. Waters, A.A. Volinsky, Gordon Research Conference, Gordon Research Conference, Thin Film and Small Scale Mechanical Behavior, Waterville, ME, 7/04 (poster),
2. *Single Crystal Surface Reconstruction Patterns*, A.A. Volinsky, M. Pendergast, B. Such, M. Szymonski, Gordon Research Conference, Thin Film and Small Scale Mechanical Behavior, Waterville, ME, 7/04 (poster),
3. *Mechanical Aspects of Anti-Corrosive Coatings Performance Tests*, A.A. Volinsky, NACE International Corrosion 2006 Conference, San Diego, CA, 3/06
4. *Moisture-induced Thin Film Adhesion Degradation*, A.A. Volinsky, P. Waters, NACE International Corrosion 2006 Conference, San Diego, CA, 3/06
5. *Microchannel Manufacturing for Lab-on-a-chip Applications*, A.A. Volinsky, 2006 US-Japan Young Researchers Exchange Program Workshop for Nanotechnology and Nanomanufacturing, Boston, MA, 3/06
6. *Moisture and Stress Effects on Thin Film and Coating Adhesion*, A.A. Volinsky, P. Waters, Army Corrosion Summit 2006, Clearwater, FL, 2/06
7. *A New Method of Testing Thin Film Adhesion in a Wet Environment*, P. Waters, A.A. Volinsky, Tri-service Corrosion Conference, Orlando, FL, 11/05 (poster award)
8. *Novel Adhesion Test for Environmentally Assisted Fracture in Thin Films*, A.A. Volinsky and P.J. Waters, Tri-service Corrosion Conference, Orlando, FL, 11/05
9. *Moisture Effects on Copper Thin Film Adhesion*, A.A. Volinsky, P.J. Waters, 2005 ASME Mechanical Engineering Congress, Orlando FL, 11/05
10. *Probing Thin Film Mechanical Properties by Nanoindentation*, A.A. Volinsky, Technische Universität Dresden, Institut für Strukturphysik, Dresden, Germany, 8/05 (invited seminar)
11. *Multidisciplinary Field of Materials Science and Engineering*, Jagiellonian University, A.A. Volinsky, Department of Physics, Astronomy and Computer Science, Krakow, Poland, 6/05 (invited lecture)
12. *Nanoindentation for Thin Film Fracture Testing*, A.A. Volinsky, NanoPol 2005:Frontiers of Nanomechanical Testing Workshop, Krakow, Poland, 6/05
13. *Bridging Thin Film Fracture and Surface Science*, A.A. Volinsky, Jagiellonian University, Department of Physics, Astronomy and Computer Science, Krakow, Poland, 5/05 (invited seminar)
14. *Sub-Critical Telephone Cord Delamination Propagation,* P. Waters, NACE International Corrosion 2005 Conference, Houston, TX, 3/05
15. *Stress-Induced Periodic Fracture Patterns in Thin Films*, A.A. Volinsky, N.R. Moody, D.C. Meyer, 11th International Congress on Fracture, Turin, Italy, 3/05
16. *Sub-Critical Telephone Cord Delamination Propagation and Adhesion Measurements*, Materials Research Society 2004 Fall Meeting, A.A. Volinsky, P.J. Waters, J.D. Kiely, E.C. Johns, Stability of Thin Films and Nanostructures Symposium, Boston, MA, 12/04
17. *Micro-fluidics Applications of Telephone Cord Delamination Blisters*, A. A. Volinsky, P.J. Waters, G. Wright, *(Poster)*, Materials Research Society 2004 Fall Meeting, Mechanically Active Materials Symposium, Boston, MA, 12/04
18. *Thin Film Mechanical Reliability: Environmental Effects*, A.A. Volinsky, NIST Workshop on Reliability Issues in Nanomaterials, Boulder, CO, 8/04

The following papers were published acknowledging NACE support (attached to the report):

1. *Stress and Moisture Effects on Thin Film Buckling Delamination*, P. Waters, A.A. Volinsky, Experimental Mechanics, 2006
2. *Novel Adhesion Test for Environmentally Assisted Fracture in Thin Films*, A.A. Volinsky and P. Waters, Tri-Service Conference on Corrosion, November 14-18, 2005, Orlando, FL, Proceedings Paper 104, 2005
3. *Mechanical Aspects of Anti-Corrosive Coatings Performance Tests*, A.A. Volinsky, NACE International Corrosion 2006 Conference Proceedings, Paper 06041, March 12-16, San Diego, CA, 2005
4. *Moisture Effects on Copper Thin Film Adhesion*, P.J. Waters, A.A. Volinsky, Proceedings of IMECE: ASME 2005 Orlando FL, Nov. 5-11, paper 81532, 2005
5. *Micro-fluidics Applications of Telephone Cord Delamination Blisters*, A.A. Volinsky, P. Waters, G. Wright, Mat. Res. Soc. Symp. Proc. Vol. 855E, W3.16, 2004
6. *Sub-Critical Telephone Cord Delamination Propagation and Adhesion Measurements*, A.A. Volinsky, P. Waters, J.D. Kiely, E. Johns, Mat. Res. Soc. Symp. Proc. Vol. 854E, U9.5, 2004